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Compiled by

Kevin Flynn

Chemistry Division

April 1975

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RADIOCHEMICAL PROCEDURES AND TECHNIQUES

Compiled by

Kevin Flynn

ABSTRACT

This report constitutes a summary of the radiochemical procedures and techniques currently in use by the Chemistry Division Nuclear Chemistry Group at Argonne National Laboratory for the analysis of radioactive samples.

ARSENIC

Introduction

Arsenic is separated from fission products by the distillation of arsine (AsH_3) followed by precipitation of arsenic as the metal using sodium hypophosphite.

Procedure

Step 1. Add to the sample (free of nitrate) in a distillation flask, 3 ml of standardized arsenic carrier (~ 10 mgs As/ml). Then add 1 ml of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ solution (10 mgs/ml), 0.5 ml of SnCl_2 solution (80% SnCl_2) and 5 ml of 6 N H_2SO_4 . Add a small quantity (~ 0.1 g) of granulated zinc and sweep the AsH_3 into 20 ml of 6 N HCl containing a few drops of liquid bromine. An air stream of about $1 \text{ cm}^3/\text{sec}$ flow rate for 5 min. is adequate to quantitatively remove the AsH_3 from solution.

Step 2. Add a few drops of hydrazine solution (about 1 M) to the distillate and boil to destroy the excess bromine. Add sodium hypophosphite (~ 1 gram of solid) and boil to precipitate arsenic metal. Filter through a weighed filter disc wash with alcohol and dry at 110°C for 10 min. Cool, weigh and mount.

Notes

1. The presence of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ and SnCl_2 in the distillation flask inhibits the formation of antimony hydride. If additional antimony decontamination is necessary, precipitate arsenic as the sulfide from strong (~ 6 N) HCl .

2. Arsenic sulfide can be dissolved in either concentrated NH_4OH (15 N) or a concentrated mixture of H_2SO_4 and HNO_3 .

3. Arsenic metal can be dissolved in either HNO_3 (15 N) or HCl (12 N) containing a few drops of hydrogen peroxide.

4. If there is an excessive amount of Te activity present, additional decontamination can be achieved by precipitation of tellurium metal from the distillate with SO_2 . The SO_2 must be boiled out of the solution before adding hypophosphite.

5. The zinc metal should be combined with the solution in a closed system since the arsine is generated very rapidly after contact with the zinc. For best results the solution should be added dropwise in a closed system to the hot zinc metal.

BROMINE AND IODINE

Introduction

Bromine and iodine are separated from fission products by selective extraction into carbon tetrachloride, followed by reduction and back extraction into aqueous solution (Ref. 2).

Procedure

Step 1. Dissolve the sample in $\text{NaOH} \cdot \text{Na}_2\text{CO}_3$ solution ($\sim 2 \text{ M}$) containing 3 ml each of I^- and Br^- standardized carrier solution ($\sim 10 \text{ mgs/ml}$ of each).

Step 2. Transfer to a separatory funnel, add 10 ml of carbon tetrachloride (CCl_4), one ml of 5% NaOCl , and acidify with conc. HNO_3 .

Step 3. Add 3 ml of 1 $\underline{\text{M}}$ $\text{NH}_2\text{OH} \cdot \text{SO}_4$ solution and extract the iodine into the CCl_4 . Separate the phases and reserve the aqueous phase for the bromine determination.

Iodine

Step 4. Equilibrate the CCl_4 phase from step 3 with 10 ml of H_2O containing a few drops of 1 $\underline{\text{M}}$ NaHSO_3 until colorless. Discard the CCl_4 .

Step 5. Add to the aqueous phase 10 ml of CCl_4 , one ml of 6 $\underline{\text{M}}$ HNO_3 and a few drops of 1 $\underline{\text{M}}$ NaNO_2 . Extract the I_2 into the CCl_4 and discard the aqueous phase.

Step 6. Repeat step 4.

Step 7. Add one ml of 6 N HNO_3 to the aqueous phase and heat to remove the SO_2 . Add 2 ml of 0.1 M PdCl_2 solution and digest the PdI_2 precipitate. Filter through a weighed filter disc, wash with alcohol and dry at 110°C for 10 min. Cool, weigh and mount.

Bromine

Step 4. Add 10 ml of CCl_4 to the aqueous phase from step 3, and sufficient KMnO_4 to maintain a permanent purple color. Extract the Br_2 into the CCl_4 and discard the aqueous phase.

Step 5. Equilibrate the CCl_4 phase with 10 ml of H_2O containing 3 ml of 1 M $\text{NH}_2\text{OH}\cdot\text{SO}_4$ until colorless and discard the CCl_4 phase.

Step 6. Repeat step 4 (Bromine).

Step 7. Equilibrate the CCl_4 phase with 10 ml of H_2O containing a few drops of 1 M NaHSO_3 and discard the CCl_4 phase.

Step 8. Add 1 ml of 6 M HNO_3 , heat to remove SO_2 , and add barium to precipitate any sulphate present. Centrifuge BaSO_4 and discard the precipitate. Add 2 ml of 0.1 M AgNO_3 and digest the AgBr precipitate. Filter through a weighed filter disc, wash with alcohol and dry at 110°C for 10 min. Cool, weigh and mount.

Notes

1. PdI_2 can be dissolved by warming with conc. NH_4OH .
2. Determination of the isotopes of iodine (6.7 hr ^{135}I , 20.8 hr ^{133}I and 8.05 d ^{131}I) requires dissolution of the PdI_2 , boiling out the Xe daughters and reprecipitation and mounting of the PdI_2 . These steps are best performed after 3 days for the 20.8 hr ^{133}I determination and again after 8 days for the 8.05 d ^{131}I determination.
3. The presence of the 2.4 hr ^{132}I in equilibrium with the 78 hr ^{132}Te must be considered in the evaluation of iodine decay data. If sufficient time is allowed (about 7 hrs) between the tellurium, iodine separation and the final xenon removal the effect of this isotope is negligible.

BARIUM AND STRONTIUM

Introduction

Barium and strontium are separated from fission products by successive nitrate precipitations followed by several iron hydroxide scavengings. The barium and strontium are separated from each other by precipitation of barium as the chromate (Ref. 3).

Procedure

Step 1. Add to the sample, three ml each (10 mgs/ml) of barium and strontium standardized carrier solution and 30 ml of fuming nitric acid. Centrifuge and decant.

Step 2. Dissolve the nitrate precipitate in 2 ml of H_2O and reprecipitate with 30 ml of fuming nitric acid.

Step 3. Dissolve the precipitate in 10 ml of H_2O , add 3 mgs of iron carrier and precipitate $Fe(OH)_3$ with two ml of NH_4OH . Centrifuge and discard the precipitate.

Step 4. Repeat the $Fe(OH)_3$ precipitation by adding 3 mgs of iron carrier to the basic solution three times, centrifuging and discarding the $Fe(OH)_3$ each time.

Step 5. Neutralize the supernate with 6 M HNO_3 , add 1 ml of 6 M acetic acid and 2 ml of 6 M ammonium acetate. Heat solution to boiling and add 1 ml of 1.5 M sodium chromate solution. Centrifuge the barium chromate precipitate and reserve the supernate for the determination of strontium.

Barium

Step 6. Wash the barium chromate precipitate from step 5 with 10 ml of H_2O and dissolve it in 2 ml of 6 M HCl.

Step 7. Add 15 ml of HCl-ether reagent (5 parts conc. HCl to 1 part diethyl ether), cool, centrifuge and discard the supernate.

Step 8. Dissolve the precipitate in 10 ml of H_2O and add 10 ml of 6 N ammonium hydroxide-ammonium carbonate reagent. Filter the precipitate through a weighed filter disc, wash with alcohol and dry at $110^{\circ}C$ for 10 min. Cool, weigh and mount.

Strontium

Step 6. Add to the supernate from step 5, sufficient NH_4OH to make the solution basic and 10 ml of 6 M ammonium hydroxide-ammonium carbonate reagent. Centrifuge and discard the supernate.

Step 7. Dissolve the precipitate in 2 ml of 6 N HCl, add 10 ml of 6 M ammonium hydroxide-ammonium carbonate reagent, filter the precipitate through a weighed filter disc, wash with alcohol and dry at $110^{\circ}C$ for 10 min. Cool, weigh and mount.

Notes

If there is a large excess of barium activity over strontium activity it may be necessary to add a few mgs of barium to the supernate in step 6 and scavenge a second time with barium chromate.

ZIRCONIUM

Introduction

Zirconium is separated from fission products by lanthanum fluoride scavengings followed by successive barium fluorozirconate precipitations and a final precipitation of zirconium as the tetramandelate (Ref. 4).

Procedure

Step 1. Add to the sample 1 ml of standardized zirconium carrier solution (~ 10 mgs/ml) and adjust the acidity to 5 M in HNO_3 . Add 1 ml of $\text{NH}_2\text{OH}\cdot\text{HCl}$ if necessary to reduce Np and Pu in order that they will carry on the lanthanum fluoride scavengings. Add 1 ml HF.

Step 2. Add 5 mgs of lanthanum carrier, centrifuge and discard the precipitate.

Step 3. Repeat step 2.

Step 4. Add 2 ml of saturated barium nitrate and allow 5 min. for the barium fluorozirconate to precipitate. Centrifuge and discard the supernate.

Step 5. Dissolve the precipitate by boiling with 10 ml of boric acid and 1 ml of conc. HNO_3 .

Step 6. Add excess NH_4OH , centrifuge and dissolve precipitate in 10 ml of 5 N HNO_3 .

Step 7. Repeat steps 4, 5 and 6 twice.

Step 8. Add to the final solution from step 7 a few drops of conc. H_2SO_4 and centrifuge. Discard the BaSO_4 precipitate.

Step 9. Add excess NH_4OH to the supernate from step 8, centrifuge and discard the supernate.

Step 10. Wash the precipitate with 10 ml of H_2O containing a few drops of NH_4OH and then dissolve it in 2 ml of conc. HCl .

Step 11. Dilute to 10 ml, heat to boiling and add 10 ml of 16% mandelic acid solution. Boil to coagulate the precipitate, filter through a weighed filter disc, wash with alcohol and dry at 110°C for 10 min. Cool, weigh and mount.

Notes

1. The zirconium tetramandelate precipitates more readily at elevated temperature so it is necessary to keep the solution hot for several minutes to get quantitative formation.

2. The edges of the filter paper must be washed thoroughly before drying. The presence of traces of the mandelic acid reagent will cause the paper to char at 110°C .

3. During the precipitation of barium fluorozirconate the solution should be maintained at 3 N in acid or greater to prevent the precipitation of barium fluoride.

MOLYBDENUM

Introduction

Molybdenum is separated from fission products by extraction into diethyl ether from 6 N HCl followed by back extraction into H₂O (Ref. 5).

Procedure

Step 1. Add to the sample 1 ml of standardized molybdenum carrier solution (~10 mgs/ml), 1 ml of tellurium carrier solution (~10 mgs/ml), a few mgs of iron carrier and adjust the acidity to 6 N HCl (add a few drops of bromine water to ensure oxidation of molybdenum to the 6+ state).

Step 2. Extract the molybdenum into 50 ml of diethyl ether which has been pre-equilibrated with 6 N HCl. Discard the aqueous phase.

Step 3. Wash the ether phase twice with 5 ml of 6 N HCl and discard washings.

Step 4. Back extract the molybdenum with two 10 ml portions of H₂O. Discard the ether phase and heat the aqueous phase briefly to remove the ether.

Step 5. Add NH₄OH in excess to precipitate the Fe(OH)₃. Centrifuge, discard the precipitate, add 3 mgs of iron carrier and again centrifuge and discard the precipitate.

Step 6. Make the solution just acid with HCl using methyl orange indicator. Add 1 ml of 6 M sodium acetate and precipitate the molybdenum by adding 1 ml of 5% 8-hydroxyquinoline in 1 M HCl. Filter through a weighed filter disc, wash with alcohol and dry at 110°C for 10 min. Cool, weigh and mount.

Notes

1. For best results with ^{99}Mo , count sample through a 15 mg/cm² aluminum absorber to absorb out the weaker radiations emitting from the sample.
2. In cases where there is excessive contamination from thorium activity, further purification can be obtained by selectively precipitating molybdenum as the α -benzoinoxine.

RUTHENIUM

Introduction

Ruthenium is separated from fission products by distillation of ruthenium tetroxide from perchloric acid followed by precipitation of ruthenium metal (Ref. 6).

Procedure

Step 1. Add to the sample in a distillation flask, 3 ml of standardized ruthenium carrier solution (~ 10 mgs/ml), 0.1 g of sodium bismuthate, 1 ml of phosphoric acid and 10 ml of perchloric acid.

Step 2. Heat the solution to fumes of perchloric and distill the ruthenium tetroxide into 15 ml of 6 N sodium hydroxide. Continue distillation until the perchloric acid is colorless.

Step 3. Add 2 mgs of iron carrier to the distillate, centrifuge and discard the $\text{Fe}(\text{OH})_3$ precipitate.

Step 4. Add 3 ml of ethanol to the distillate, heat to boiling and centrifuge. Discard the supernate and wash the precipitate once with 10 ml of 1 N sodium hydroxide.

Step 5. Dissolve the precipitate in 2 ml of 6 N HCl, add 10 ml of H_2O and carefully add small portions of powdered magnesium metal until the solution turns black. Add 12 M HCl carefully until all the excess magnesium is dissolved. Centrifuge, discard the supernate and wash the precipitate thoroughly to remove perchlorate salts.

Step 6. Filter through a weighed filter disc, wash thoroughly with H_2O and alcohol and dry at $110^{\circ}C$ for 10 min. Cool, weigh and mount.

Notes

1. If the precipitate is not thoroughly washed free of perchlorate salts, the filter paper will char when heated.
2. Ruthenium precipitated by magnesium reduction weighs about 6% higher than ruthenium made by hydrogen reduction. If the carrier is standardized by magnesium reduction, this error cancels.
3. Ruthenium precipitated by magnesium reduction can be dissolved in 12 M sodium hydroxide containing sodium hypochlorite.

RHODIUM

Introduction

Rhodium is separated from fission products by precipitation of the triethallium hexanitrorhodate, followed by thallium oxide scavengings and final precipitation of the hexa-amminecobalt hexanitrorhodate (Ref. 7).

Procedure

Step 1. Add to the sample in nitric acid 1 ml of standardized rhodium carrier solution (~ 10 mgs/ml), and 1 mg of palladium carrier. Dilute solution to 1 N in HNO_3 . Add 0.3 ml of 1 M KMnO_4 and warm gently. Reduce the excess oxidant by dropwise addition of saturated aqueous SO_2 .

Step 2. Add 1 ml of saturated NaNO_2 solution and warm gently. Add 0.1 g of solid TlNO_3 and stir well. Centrifuge, discard the supernate and wash the precipitate with 10 ml of 6 N HCl .

Step 3. Dissolve the precipitate by boiling with 1 ml of aqua regia. Dilute to 10 ml with water.

Step 4. Repeat steps 2 and 3.

Step 5. Add 6 M NaOH dropwise to the final solution from step 4 until the hydrated Tl_2O_3 precipitate is well coagulated. Centrifuge and discard the precipitate.

Step 6. Add 2 drops of methyl orange indicator acidify the supernate from step 5 with 6 N HCl . Add 2 ml of saturated NaNO_2 followed by 3 ml of saturated $\text{Co}(\text{NH}_3)_6\text{Cl}_3$ solution. Warm gently. Filter through a weighed filter disc, wash with alcohol, dry at 110°C for 10 min. Cool, weigh and mount.

Notes

Alternatively the purified rhodium from step 5 may be precipitated as the metal by diluting to 20 ml of 3 N HCl and carefully adding finely divided magnesium metal (about 0.3 g). Boil to complete the reduction, dissolve the excess magnesium with concentrated HCl, filter through a weighed filter disc, wash with alcohol, dry at 110° for 10 min. cool, weigh and mount. If rhodium is to be determined as the metal, 3 ml of standardized rhodium carrier solution should be added in step 1.

PALLADIUM

Introduction

Palladium is separated from fission products by precipitation as the dimethylglyoxime coupled with iron hydroxide scavengings from ammoniacal solution (Ref. 8).

Procedure

Step 1. Add to the sample 1 ml of standardized palladium carrier solution (~10 mgs/ml), adjust the acidity to 0.4 N in HCl and add 3 ml of 1% dimethylglyoxime in ethanol. Centrifuge, discard the supernate and wash the precipitate with 10 ml of H₂O.

Step 2. Dissolve the precipitate in 1 ml of hot HNO₃ (boil vigorously to destroy the dimethylglyoxime), dilute to 10 ml with H₂O, add 3 mgs of iron carrier and make basic with excess NH₄OH. Add 10 mgs of silver carrier and just enough iodide carrier to precipitate all the silver. Centrifuge and discard the precipitate.

Step 3. Add 3 mgs of iron carrier and 10 mgs of silver carrier to the supernate. Centrifuge and discard the precipitate.

Step 4. Acidify the supernate to 0.4 M in HCl, centrifuge and discard the silver chloride precipitate.

Step 5. Add to the supernate, 3 ml of 1% dimethylglyoxime solution. Filter through a weighed filter disc, wash with alcohol and dry at 110° for 10 min. Cool, weigh and mount.

Notes

1. For best results count palladium fission product samples through a 15 mg/cm^2 aluminum absorber in order to absorb the weaker radiations emitting from the sample. This absorbs the ^{112}Pd parent as well as the I.T. daughter of the ^{109}Pd .

2. The 13.5 hr ^{109}Pd - 21.0 hr ^{112}Pd mixture can best be resolved with a "Biller" plot (Ref. 15) of the counting data. See Appendix B.

SILVER

Introduction

Silver is separated from fission products by precipitation of silver chloride, iron hydroxide scavengings from ammoniacal solution and precipitation of silver sulfide (Ref. 9).

Procedure

Step 1. Add to the sample, three ml of standardized silver carrier solution (~ 10 mgs/ml), 1 ml of 6 N HCl and about 20 ml of water. Centrifuge, decant and wash the precipitate with 10 ml of water.

Step 2. Dissolve the AgCl precipitate in 2 ml of concentrated NH_4OH , dilute to 15 ml, add a few drops of ammonium carbonate solution and 3 mgs of iron carrier. Centrifuge and discard the precipitate. Add another 3 mgs of iron, centrifuge and again discard the precipitate.

Step 3. Add 3 ml of ammonium sulfide solution to the supernate from step 2, centrifuge and discard the supernate.

Step 4. Dissolve the Ag_2S by heating with 1 ml of concentrated HNO_3 , dilute to 15 ml, add excess NH_4OH , 3 mgs of iron carrier, centrifuge and discard the precipitate. Add another 3 mgs of iron, centrifuge and again discard the precipitate.

Step 5. Add 3 ml of ammonium sulfide solution to the supernate from step 4, centrifuge and discard the supernate.

Step 6. Dissolve the Ag_2S by heating with 1 ml of concentrated HNO_3 , dilute to 15 ml, neutralize with concentrated NH_4OH and add 2 ml in excess. Add 3 mgs of iron carrier, centrifuge and discard the precipitate.

Step 7. Neutralize the supernate from step 6 with HNO_3 , add 2 ml of 6 N HCl and immediately filter through a weighed filter disc. Wash with alcohol, dry at 110°C for 10 min. Cool, weigh and mount.

Notes

1. Immediate filtration of the final AgCl precipitate will result in a uniform spread over the filter disc. If the precipitate is allowed to stand before filtering it will coagulate into large clumps.

2. If the fission yield of ^{113}Ag is being determined it must be remembered that a substantial fraction (about 30%) decays through the 1.2 min isomer.

CADMIUM

Introduction

Cadmium is separated from fission products by precipitation of the sulfide from dilute acid followed by basic iron acetate scavenges and palladium sulfide scavenges from strong acid (Ref. 10).

Procedure

Step 1. Add to the sample 3 ml of standardized cadmium carrier solution (~ 10 mgs/ml). Evaporate just to dryness, take up in 20 ml of 0.2 N HCl, gas the solution with H_2S , centrifuge and discard the supernate.

Step 2. Dissolve the precipitate in 1 ml of concentrated HCl, dilute to 20 ml, add 3 mgs of iron carrier and precipitate $Fe(OH)_3$ by adding excess NH_4OH . Centrifuge and discard the precipitate.

Step 3. Add 3 mgs of iron carrier and 6 N HCl dropwise until precipitate dissolves. Heat to boiling and add about 5 drops of 6 M ammonium acetate. Continue boiling until precipitate is well coagulated. Centrifuge and discard the precipitate.

Step 4. Make the solution just acid with HCl, gas with H_2S , centrifuge and discard the supernate.

Step 5. Repeat steps 2, 3 and 4.

Step 6. Dissolve the CdS in 1 ml of concentrated HCl, add 10 ml of 6 N H_2SO_4 , 2 mgs of Pd and gas with H_2S . Centrifuge and discard the precipitate.

Step 8. Neutralize the supernate with NH_4OH until it is just acid, gas with H_2S , centrifuge and discard the supernate.

Step 9. Dissolve the CdS precipitate in 1 ml of concentrated HCl , take just to dryness and dilute to 20 ml with H_2O . Add NH_4OH to basic methyl red, add 3 ml of 3 M ammonium chloride, heat to boiling and add 2 ml of 1.5 M dibasic ammonium phosphate. Filter through a weighed filter disc, wash with alcohol, dry at 110°C for 10 min. Cool, weigh and mount.

Notes

If zinc is a possible contaminant be sure the CdS is precipitated from $>0.1 \text{ N}$ HCl (zinc precipitates from $.01 \text{ N}$ acid). Alternatively zinc and cadmium can be separated by TBP extraction. Zinc will extract from a 3 M NaCl solution into TBP leaving cadmium in the aqueous phase.

TIN

Introduction

Tin is separated from fission products by precipitation of the sulfide from 2 N HCl, followed by sulfide scavenges from HF solution and iron scavenges from strong NaOH solution.

Procedure

Step 1. Add to the sample 2 ml of standardized tin carrier solution (10 mgs/ml), one drop of bromine and sufficient HCl to make the final solution 2 N in acid. Boil out the bromine, gas with H_2S , centrifuge and discard the supernate.

Step 2. Dissolve the SnS_2 precipitate in 2 ml of concentrated HCl. Add 5 mgs of antimony carrier (trivalent), 1 ml of concentrated H_2SO_4 , 1 ml of concentrated HF and dilute to 15 ml. Add 5 mgs of lanthanum carrier and gas with H_2S . Centrifuge and discard the precipitate.

Step 3. Add another 5 mgs of antimony and 5 mgs of lanthanum, gas with H_2S , centrifuge and discard the precipitate.

Step 4. Repeat step 3.

Step 5. Add 10 ml of saturated boric acid to the supernate from step 4, gas with H_2S , centrifuge and discard the supernate.

Step 6. Dissolve the SnS_2 precipitate in 2 ml of concentrated HCl, dilute to 15 ml, heat, and add 12 M NaOH until the $Sn(OH)_4$ redissolves. Add 2 drops of Na_2CO_3 solution, 3 mgs of iron carrier, centrifuge and discard the precipitate.

Step 7. Add 3 mgs of iron, centrifuge and discard the precipitate.

Step 8. Repeat step 7.

Step 9. Acidify the supernate from step 8 with concentrated H_2SO_4 (add acid carefully). Gas with H_2S , centrifuge and discard the supernate.

Step 10. Dissolve the SnS_2 precipitate in 2 ml of concentrated HCl , dilute to 15 ml of 2 N HCl , gas with H_2S , centrifuge and discard the supernate.

Step 11. Dissolve the SnS_2 precipitate in 2 ml of concentrated HCl and dilute to 10 ml of 3 N HCl . Add 1 ml of 5% 8-hydroxy-quinolate in 1 N HCl and then add 6 M ammonium acetate dropwise until precipitate forms permanently. Add 2 drops excess, filter through a weighed filter disc, wash with alcohol and dry at 110°C for 10 min. Cool, weigh and mount.

Notes

Alternatively the tin may be precipitated for mounting as the metal by adding 3 ml of hot chromous chloride solution (oxsorbent) to the tin contained in 10 ml of hot 1 N HCl solution.

ANTIMONY-1

Introduction

Antimony is separated from fission products by precipitation as the sulfide from 4 N HCl, iron scavengings from strong NaOH solution and specific removals of molybdenum and ruthenium.

Procedure

Step 1. Add to the sample, 3 ml of standardized antimony carrier (~10 mgs/ml), one drop of bromine, 10 mgs of molybdenum carrier, and sufficient HCl to make 15 ml of 3 N HCl. Boil out the bromine, gas with H_2S , centrifuge and discard the supernate.

Step 2. Leach the precipitate with 5 ml of concentrated HCl (molybdenum sulfide does not dissolve), dilute to 10 ml, centrifuge and discard the precipitate. Dilute to 3 N in HCl, gas with H_2S , centrifuge and discard the supernate.

Step 3. Dissolve the precipitate in concentrated HCl and centrifuge out any insoluble residue. Dilute to 10 ml with H_2O , heat to boiling and add 10 N NaOH until the antimony redissolves. Add a few drops of Na_2CO_3 solution, 3 mgs of iron carrier, centrifuge and discard the precipitate.

Step 4. Add 3 mgs of iron carrier, centrifuge and discard the precipitate.

Step 5. Repeat step 4.

Step 6. Acidify the supernate from step 5 with concentrated H_2SO_4 (add H_2SO_4 carefully), gas with H_2S , centrifuge and discard the supernate.

Step 7. Dissolve the precipitate in concentrated HCl, dilute to 3 N in HCl, gas with H_2S , centrifuge and discard the supernate. Dissolve the precipitate in concentrated HCl.

Step 8. Make the solution up to about 5 ml of 4 N HCl, add 5 ml of freshly prepared chromous chloride solution and let stand for 10 min. Filter the Sb^0 through a weighed filter disc, wash thoroughly with alcohol and dry at $110^\circ C$ for 10 min. Cool, weigh and mount.

Notes

1. Chromous chloride solution is prepared fresh by boiling a 1 M solution of $CrCl_3$ in 3 N HCl with fine grain zinc metal. Excess zinc metal can be centrifuged out.

2. Alternatively molybdenum can be specifically removed by precipitation as the α -benzoinoxime.

3. If ruthenium is present in excessive amounts it can be removed by fuming with $HClO_4$ in the presence of ruthenium carrier after step 7. The resulting white antimony precipitate can be dissolved in hot concentrated H_2SO_4 . The antimony can then be precipitated as the sulfide by diluting to 6 N and gassing with H_2S .

ANTIMONY-2

Introduction

If chemical yield is relatively unimportant, antimony can be separated from fission products by distillation of stibine from sulfuric acid. The antimony yield from this distillation usually runs about 30%.

Procedure

Step 1. Add to the sample (free of nitrate) 5 ml of standardized antimony carrier solution (~ 10 mgs/ml), 4 ml of 6 N H_2SO_4 and one drop of bromine water. Add this solution dropwise in a closed distillation system to about one gram of hot granulated zinc. Sweep the stibine gas into 20 ml of 6 N HCl containing a few drops of liquid bromine. An air stream of about $1 \text{ cm}^3/\text{sec}$ flow rate for 5 min. is adequate to remove the SbH_3 from solution.

Step 2. Add 10 mgs of arsenic carrier to the distillate and just enough hydrazine HCl to destroy the excess bromine. Heat to boiling, gas with H_2S , centrifuge and discard the precipitate.

Step 3. Dilute the solution to 3 N in HCl , gas with H_2S , centrifuge and discard the supernate. Dissolve the precipitate in concentrated HCl .

Step 4. Make solution up to about 5 ml of 4 N HCl , add 5 ml of freshly prepared chromous chloride solution and let stand for 10 min. Filter the Sb^0 through a weighed filter disc, wash thoroughly with alcohol and dry at 110°C for 10 min. Cool, weigh and mount.

Notes

1. It is necessary to add the antimony solution to the zinc metal in a closed system since the stibine is generated very rapidly after contacting the zinc.

2. The precipitation of arsenic as the sulfide in step 2 represents zero time for the growth of Te daughters into the antimony isotopes.

TELLURIUM

Introduction

Tellurium is separated from fission products by precipitation of the metal from strong HCl solution followed by iron scavenges from ammoniacal solution (Ref. 11).

Procedure

Step 1. Add to the sample 3 ml of standardized tellurium carrier solution (~ 10 mgs/ml) and 10 ml of concentrated HCl. Evaporate nearly to dryness and take up in 20 ml of 3 N HCl. Heat nearly to boiling and gas with SO_2 until the tellurium metal precipitate is well coagulated. Centrifuge and discard the supernate.

Step 2. Dissolve the Te^0 precipitate in 1 ml of concentrated HNO_3 , evaporate nearly to dryness, add 1 ml of 6 N HCl and dilute to 10 ml. Add 3 mgs of palladium carrier and 3 ml of 1% dimethylglyoxime (DMG) in ethanol. Let solution stand until the Pd DMG is well coagulated. Centrifuge and discard the precipitate.

Step 3. Add another 3 mgs of palladium, let stand, centrifuge and discard the precipitate.

Step 4. Adjust the acidity of the supernate from step 3 to 3 N in HCl, heat, gas with SO_2 , centrifuge and discard the supernate.

Step 5. Dissolve the Te^0 precipitate in 1 ml of concentrated HNO_3 , evaporate nearly to dryness, add 1 ml of 6 N HCl and dilute to 10 ml. Add concentrated NH_4OH dropwise until the H_2TeO_3 precipitate redissolves. Add 10 drops excess. Add 3 mgs of iron carrier, centrifuge and discard the precipitate.

Step 6. Add 3 mgs of iron carrier, centrifuge and discard the precipitate.

Step 7. Repeat step 6.

Step 8. Add an equal quantity of 6 N HCl to the supernate from step 7, heat to boiling, gas with SO₂, centrifuge and discard the supernate.

Step 9. Slurry the Te^o precipitate with 10 ml of hot water, filter through a weighed filter disc, wash with alcohol and dry at 110°C for 10 min. Cool, weigh and mount.

Notes

1. For best results when determining ¹³²Te by beta counting, cover the sample with a 15 mg/cm² aluminum absorber. This absorbs the weaker radiation from the ¹³²Te parent and allows for a more unambiguous absorption and scattering factor correction based solely on the hard beta radiations from the ¹³²I daughter.

2. If copper is present as a contaminant it is necessary to scavenge with iron from sodium hydroxide solution (no ammonium ion) at least once.

CESIUM AND RUBIDIUM

Introduction

Cesium and rubidium are separated from fission products by precipitation of the perchlorate followed by iron hydroxide scavengings from ammoniacal solution. Cesium can then be precipitated as the bismuth iodide leaving the rubidium in solution (Ref. 12).

Procedure

Step 1. Add to the sample 3 ml of standardized cesium and/or rubidium carrier solutions (~ 10 mgs/ml). Add 5 ml of 70% HClO_4 . Add 25 ml of absolute ethanol, cool and stir. Centrifuge and discard the supernate.

Step 2. Dissolve the precipitate in 10 ml of H_2O , add 3 mgs of iron carrier, heat and add 6 N NH_4OH dropwise until the $\text{Fe}(\text{OH})_3$ precipitate is well coagulated. Centrifuge and discard the precipitate.

Step 3. Add 3 mgs of iron carrier, 1 drop of 6 N NH_4OH , centrifuge and discard the precipitate.

Step 4. Repeat step 3.

Step 5. Add 5 drops of 6 N NaOH to the supernate from step 4 and boil down to about 3 ml. Add 5 ml of 70% HClO_4 , evaporate to fumes of HClO_4 , cool and add 25 ml of absolute ethanol. Cool, stir and centrifuge. Discard the supernate and wash the precipitate twice with 10 ml portions of absolute alcohol.

If cesium or rubidium are being determined exclusively the sample can be filtered, washed with ethanol, dried at 110°C for 10 min., cooled, weighed and mounted at this point. If a cesium-rubidium separation is necessary continue with step 6.

Step 6. Dissolve the perchlorate precipitate from step 5 in 7 ml of H_2O . Add 3 ml of BiI_3 -HI reagent (10 g of BiI_3 dissolved in 50 ml of 55% HI). Centrifuge out the $\text{Cs}_3\text{Bi}_2\text{I}_9$ precipitate and wash with 10 ml of H_2O . Save the supernate for rubidium determination (see step 8).

Step 7. Dissolve the $\text{Cs}_3\text{Bi}_2\text{I}_9$ in 1 ml concentrated HNO_3 . Evaporate almost to dryness and take up in 20 ml of 0.5 N HCl . Gas with H_2S and discard the Bi_2S_3 precipitate. Evaporate the supernate to about 1 ml, add 4 ml of 70% HClO_4 and evaporate to fumes of HClO_4 . Add 25 ml of absolute ethanol and cool. Centrifuge, wash the precipitate thoroughly with absolute ethanol, filter through a weighed filter disc, wash with ethanol and dry at 110°C for 10 min. Cool, weigh and mount.

Step 8. To the supernate from step 6, add 10 mgs of cesium carrier and warm the solution gently. Centrifuge and discard the precipitate. If cesium is present in excessive amounts relative to the rubidium it may be necessary to repeat this step several times. Each cesium scavenge is worth about a factor of 50 in decontamination.

Step 9. Evaporate the supernate from step 8 to dryness and sublime off all the iodine. Take up the residue in 20 ml of 0.5 N HCl . Gas with H_2S and discard the Bi_2S_3 precipitate.

Evaporate the supernate to about 1 ml, add 4 ml of 70% HClO_4 and evaporate to fumes of HClO_4 . Add 25 ml of absolute ethanol and cool. Centrifuge, wash the precipitate thoroughly with absolute ethanol, filter through a weighed filter disc, wash with ethanol and dry at 110°C for 10 min. Cool, weigh and mount.

Notes

Be careful to wash the filter paper thoroughly with ethanol, or perchlorate salts will cause the paper to char during drying.

CERIUM

Introduction

Cerium is separated from fission products by extraction of Ce^{+4} into hexone from 10 N HNO_3 followed by back extraction of Ce^{+3} into water (Ref. 13).

Procedure

Step 1. Add to the sample 3 ml of standardized cerium carrier solution (~ 10 mgs/ml). Adjust the solution volume to about 15 ml of 10 N HNO_3 . Add 2 ml of 2 M NaBrO_3 and extract into 100 ml of hexone. (Hexone must be pre-equilibrated with 10 N HNO_3). Wash the hexone phase twice with 10 ml of 10 N HNO_3 containing a few drops of 2 M NaBrO_3 .

Step 2. Back extract the cerium with 10 ml of H_2O containing about 0.3 ml of 30% hydrogen peroxide solution.

Step 3. Neutralize the aqueous phase with concentrated NH_4OH . Add 1 ml of 6 N HNO_3 and about 15 ml of saturated oxalic acid. Centrifuge and discard the supernate.

Step 4. Dissolve the precipitate from step 3 in 1 ml of 6 N HNO_3 , dilute to 15 ml with H_2O , heat to boiling and add 15 ml of saturated oxalic acid. Centrifuge and decant.

Step 5. Slurry the precipitate from step 4 with H_2O , filter through a weighed filter disc, wash with alcohol and dry at 110°C for 10 min. Cool, weigh and mount.

Notes

1. If Pu, Np or U are present to the extent that they are serious radioactive contaminants, the hexone extraction cycle should be repeated.

2. If Th is present as a possible contaminant the 10 N HNO_3 phase should be extracted with TBP before oxidizing the cerium to the +4 state.

3. If Pa is present as a possible contaminant, it can be removed by extraction into di-isobutyl-carbinol from 6 N HCl .

RARE EARTHS

Introduction

The rare earths are separated from fission products and each other by "carrier free" elution from a di-ethyl-hexyl-ortho-phosphoric acid (HDEHP) column (Ref. 14). The rare earths thus determined are known only "relatively" to each other. They can be normalized to other fission yields via the cerium isotopes which are determined from these measurements as well as by conventional radiochemical techniques.

Procedure

Step 1. Add to the sample one mg of iron carrier solution and enough excess 10 M NaOH to redissolve all amphoteric elements. Centrifuge and discard the supernate.

Step 2. Dissolve the $\text{Fe}(\text{OH})_3$ precipitate from step 1 in 1 ml of 6 N HCl, dilute to 10 ml and again add sufficient excess 10 M NaOH to redissolve all amphoteric elements. Centrifuge and discard the supernate.

Step 3. Dissolve the $\text{Fe}(\text{OH})_3$ precipitate from step 2 in 1 ml of 6 N HCl, dilute to 20 ml with H_2O , heat solution gently and add concentrated NH_4OH dropwise until the $\text{Fe}(\text{OH})_3$ precipitates. Centrifuge, discard the supernate and wash the precipitate with 10 ml of hot H_2O .

Step 4. Dissolve the $\text{Fe}(\text{OH})_3$ precipitate in 2 ml of 6 N HCl and extract with 5 ml of di-ethyl-ether (ether must be pre-equilibrated with 6 N HCl). Separate phases and discard the ether phase.

Step 5. Add 5 ml of di-ethyl-ether to the aqueous phase from step 4 and extract. Separate the phases and discard the ether phase.

Step 6. Repeat step 5.

Step 7. Evaporate the aqueous phase from step 6 to dryness and take up residue in 0.5 ml of 0.1 N HCl. This solution is then loaded on a 10 cm long by 0.3 cm diameter column (free column volume about 0.5 ml) of HDEHP adsorbed on celite (Ref. 14). The column is run at 50°C with an elution rate of about 0.3 ml per min. A typical elution curve indicating the position of the various rare earth fractions is shown in Fig. 1. The eluate fractions taken for the various rare earths are indicated in Table 1.

Step 8. The cerium is separated from the lanthanum and praesodymium by the addition of 3 ml of standardized cerium carrier solution followed by the cerium radiochemical purification procedure (see cerium procedure). The other rare earths are coprecipitated as the oxalate with 10 mgs of standardized lanthanum carrier, filtered through a weighed filter disc, washed with alcohol, dried at 110°C for 10 min., cooled, weighed and mounted.

Notes

The 53 hr ^{149}Pm -28 hr ^{151}Pm mixture can best be resolved with a "Biller" plot (Ref. 15) of the counting data. See Appendix B.

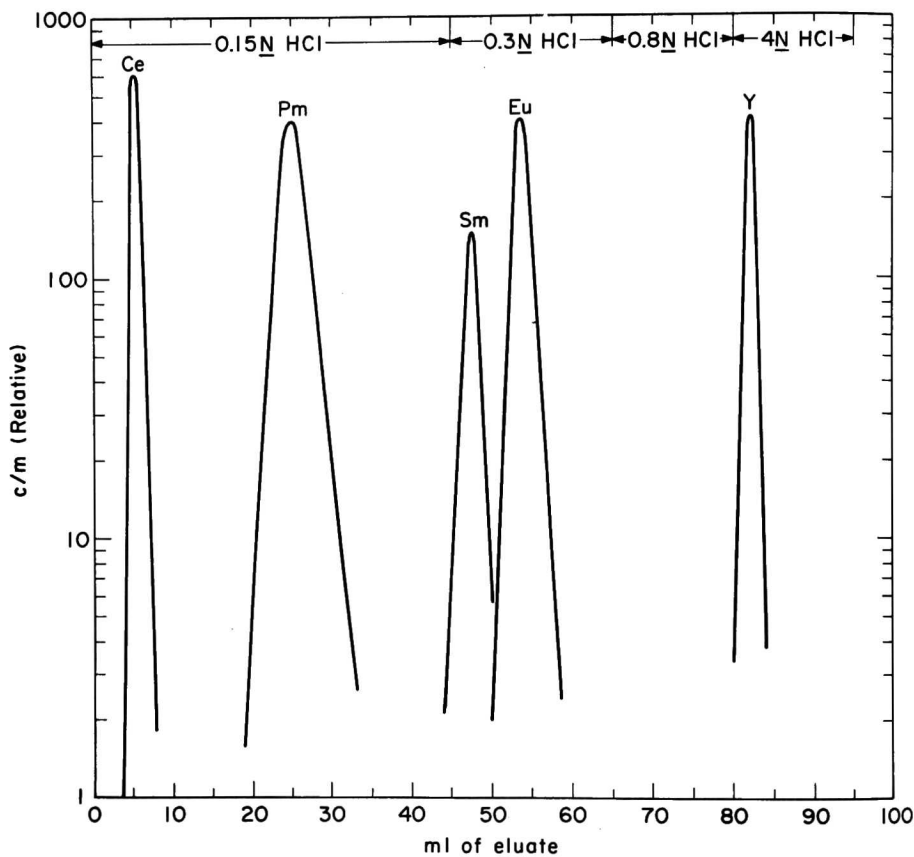


Fig. 1. Rare earth elution curve. ANL Neg. No. 122-2263.

Table 1. Rare Earth Eluate Fractions

Acid Normality (HCl)	Eluate Volume (ml)	Rare Earth Fraction
0.15	0-2	---
0.15	3-11	La,Ce,Pr
0.15	12-17	Nd
0.15	18-35	Pm
0.15	36-45 }	Sm
0.30	46-50 }	
0.30	51-60	Eu
0.30	60-65 }	Gd,Tb
0.80	65-80 }	
4.0	80-95	Y

TANDEM PROCEDURES

Introduction

In many cases it is convenient to determine several elements from one sample. To this end, the following tandem procedures for initial separations have been devised. After the initial separation the element is purified by the normal radiochemical procedure.

Procedure I: Sr, Mo, Ag, Cd, Sn, Sb, Te, Ba, Ce

After the addition of the appropriate amounts of each of the above carriers add one drop of bromine and metathasize the solution to 6 N in HCl.

Dilute Solution to 3 N in HCl \longrightarrow AgCl precipitate

↓
Add 0.5 ml concentrated H_2SO_4 \longrightarrow $BaSO_4$ and $SrSO_4$ precipitate

↓
Heat to boiling and gas with SO_2 \longrightarrow Te^0 precipitate

↓
Boil out SO_2 and gas with H_2S \longrightarrow MoS_3 and Sb_2S_3 precipitate

↓
Leach with conc. HCl \longrightarrow MoS_3 precipitate

↓
Dilute to 3 N and gas with
 $H_2S \longrightarrow Sb_2S_3$ precipitate

↓
Dilute to 1 N in HCl and gas with H_2S \longrightarrow SnS_2 precipitate

↓
Dilute to 0.1 N in HCl and gas with H_2S \longrightarrow CdS precipitate

↓
Boil out H_2S and add excess NaOH \longrightarrow $Ce(OH)_3$ precipitate.

Procedure II: Zr, Ru, Pd, Ag, Ce

After the addition of the appropriate amounts of each of the above carriers add 5 ml of concentrated H_2SO_4 , transfer to a distillation flask and evaporate to fumes of H_2SO_4 .

Add 3 ml of concentrated HClO_4 and distill $\rightarrow \text{RuO}_4$ distillate

(trap in NaOH)

↓
Evaporate to 3 ml, transfer to centrifuge tube,

dilute to 20 ml, add 1 ml of 6 N $\text{HCl} \rightarrow \text{AgCl}$ precipitate

↓
Add 1 ml of di-methylglyoxime (DMG) reagent $\rightarrow \text{Pd}$ DMG precipitate

↓
Add 1 ml of concentrated $\text{HF} \rightarrow \text{CeF}_3$ precipitate

↓
Add excess $\text{NaOH} \rightarrow \text{Zr}(\text{OH})_4$ precipitate.

APPENDIX A. Mounting Procedures

These separation-purification procedures have been designed for final mounting of a thick precipitate ($\sim 10 \text{ mg/cm}^2$) suitable for β and/or γ counting in a standardized geometry. The final precipitate is filtered through a weighed disc (2.2 cm diameter) of Whatman number 42 filter paper using a chimney arrangement as shown in Figure 2. This precipitate is then weighed to give the chemical yield. The area covered by the precipitate is about 2.5 cm^2 . The carriers used are standardized by quantitative analysis based on precipitation of the element in the same form as that used for final mounting in the specific procedure. The final precipitates are mounted on 5 cm diameter stainless steel planchets and covered with 1/4 mil (1 mg/cm^2) mylar. In those cases where alpha contamination is a serious problem, the sources are covered with an additional layer of 1 mil (4 mg/cm^2) mylar.

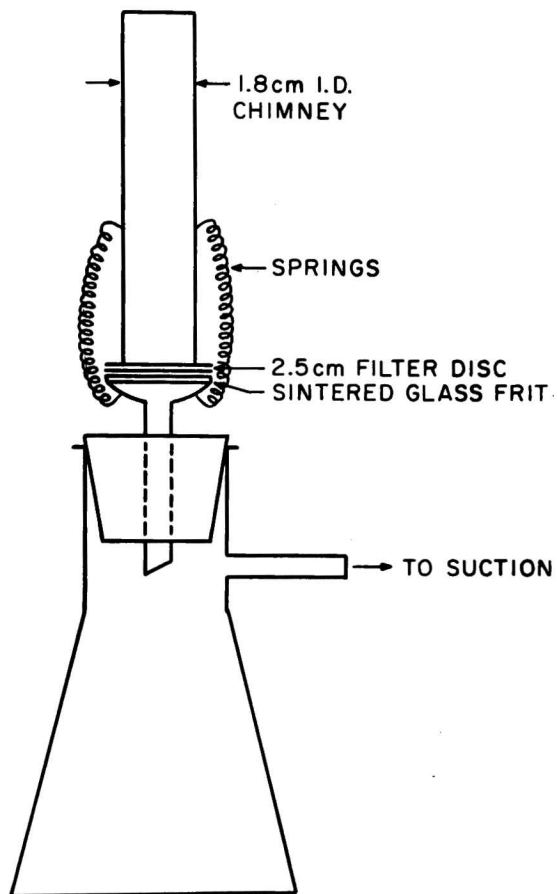
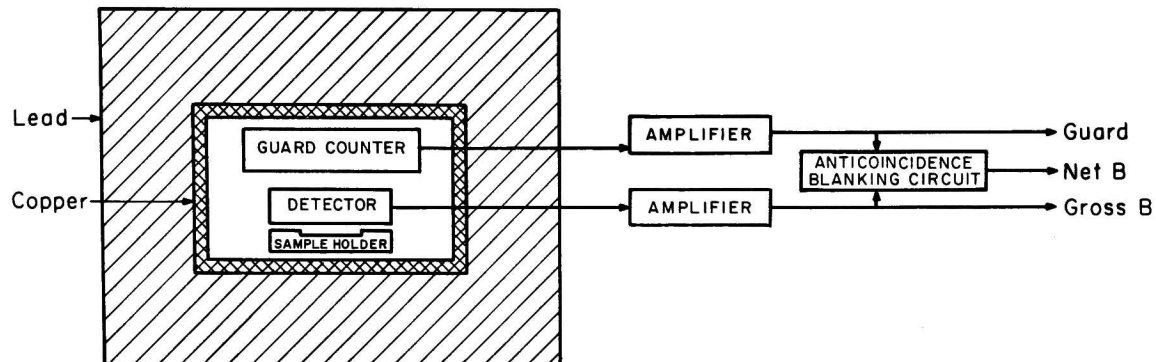


Fig. 2. Filtration apparatus. ANL Neg. No. 122-2262.

APPENDIX B. Beta Counting Procedures

β counting of the thick samples obtained from these radiochemical procedures has been done using a Sharp Laboratory model FI(P)IN, dual detector end window proportional low background system as supplied by Baird-Atomic, Inc. A brief sketch of the counter assembly with pertinent dimensions is shown in Figure 3. The counter has a background of about 0.4 c/m and a coincidence loss of about 0.5% per 10^3 c/m when operated in the "net β " mode. When operated in the "fast β " mode (i.e. no anti-coincidence cancellation) the background is about 8 c/m and the coincidence loss is about 0.6% per 10^4 c/m.

The efficiency (defined as the observed counts per minute/ $4\pi\beta$ disintegrations per minute) of the β counter for specific nuclides has been determined using standardized solutions. A carrier-free solution of the desired activity is prepared and its disintegration rate determined by an absolute beta counting technique (i.e. 4π beta counting, beta-gamma coincidence counting or standardized 2π beta counting). Aliquots of this solution are then processed using carriers and mounting procedures identical to those used in the specific radiochemical procedure. In this way the overall counting efficiency for the specific radionuclide can be determined directly. Several samples containing varying quantities of carrier were processed for each nuclide in order to establish a curve for counting efficiency (ϵ) as a function of sample weight (W). A total of 25 radionuclides and/or radionuclide



Window diameter 3.2 cm
 Sample diameter 2.0 cm
 Sample to window distance 3 mm

Fig. 3. Sharp end window proportional counter. ANL Neg. No. 122-2267.

mixtures were determined in this manner. The results of these determinations are shown in Figures 4 through 10. Pertinent information regarding counting problems unique to specific radio-nuclides is also included. For instance ^{99}Mo and ^{109}Pd are counted through a 15 mg/cm^2 aluminum absorber in order to range out the weaker radiations from the daughter nuclides. The efficiency curve for ^{103}Ru is subject to some ambiguity because of the uncertainty in the counting efficiency for the ^{103}Rh daughter.

In those cases where parent-daughter combinations are counted in equilibrium (i.e. ^{90}Sr - ^{90}Y , ^{95}Zr - ^{95}Nb , ^{106}Ru - ^{106}Rh , ^{115}Cd - ^{115}In , ^{125}Sb - ^{125}Te , ^{137}Cs - ^{137}Ba , ^{140}Ba - ^{140}La , and ^{144}Ce - ^{144}Pr) the relative activities of the parent and daughter at equilibrium have been indicated. From the known energy and the measured counting efficiency of each of these beta emitters a series of curves of counting efficiency versus energy for a constant weight of precipitate were established. These curves are shown in Figures 11 and 12.

The primary standardizations were done using 4π beta counting and beta-gamma coincidence counting techniques as described by G. D. O'Kelley (Ref. 16). A secondary standardization technique using a 2π beta counter was established for convenience. An aliquot of a solution that had been previously standardized by 4π beta and/or beta-gamma coincidence counting was evaporated on a platinum disc. This disc was then counted in a 2π beta proportional counter in order to establish the counting efficiency

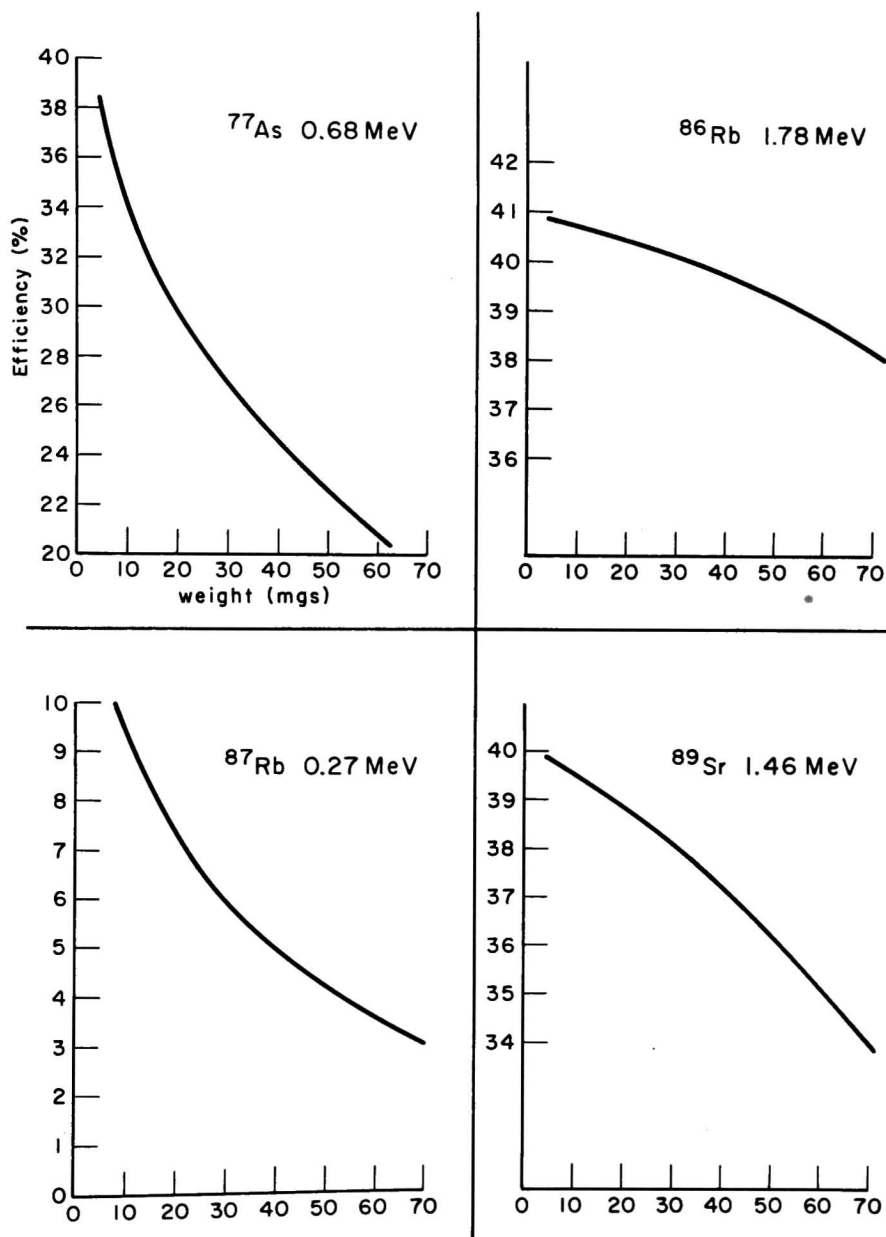
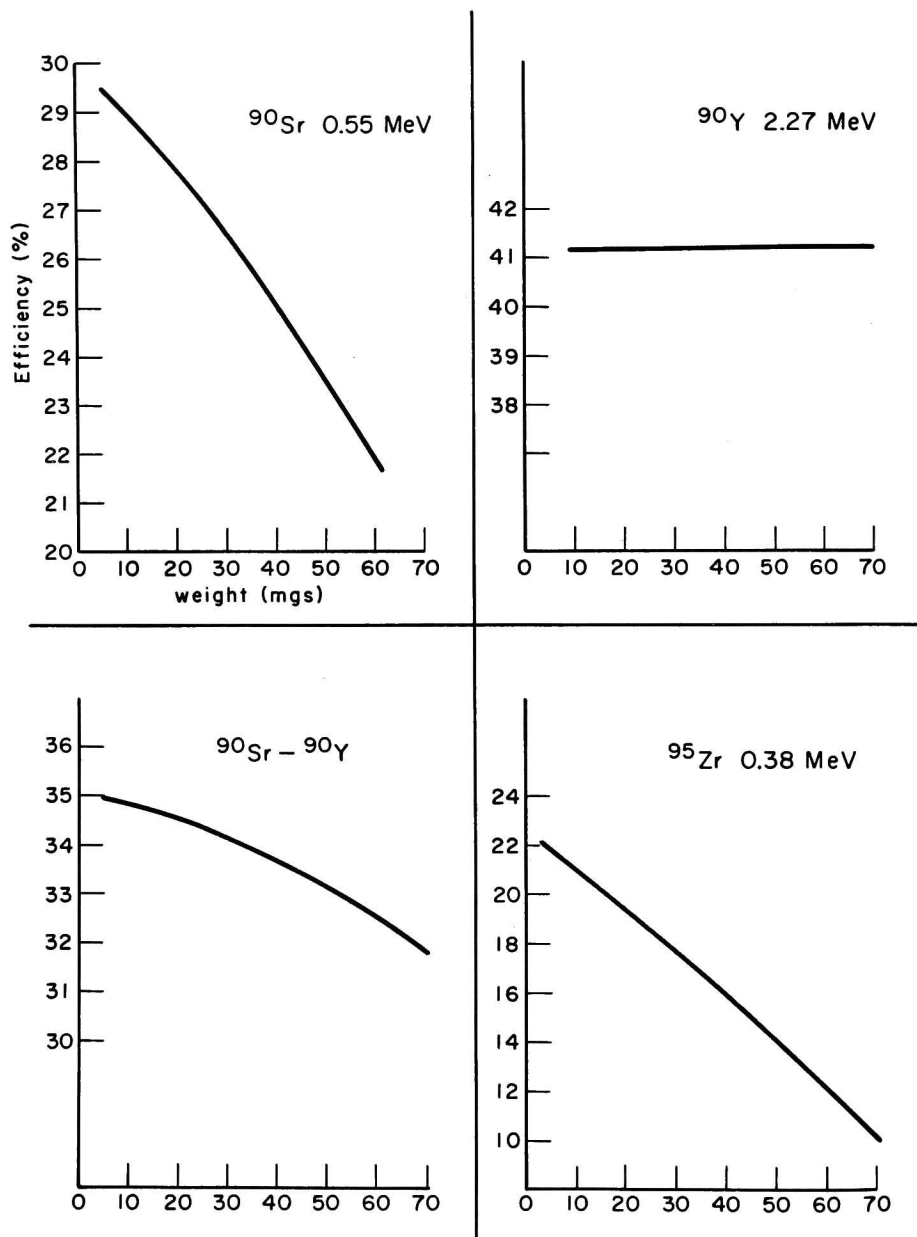


Fig. 4. β counting efficiency vs weight of precipitate. ANL Neg. No. 122-2266.

Fig. 5. β counting efficiency vs weight of precipitate, ANL Rep. No. 199-0071

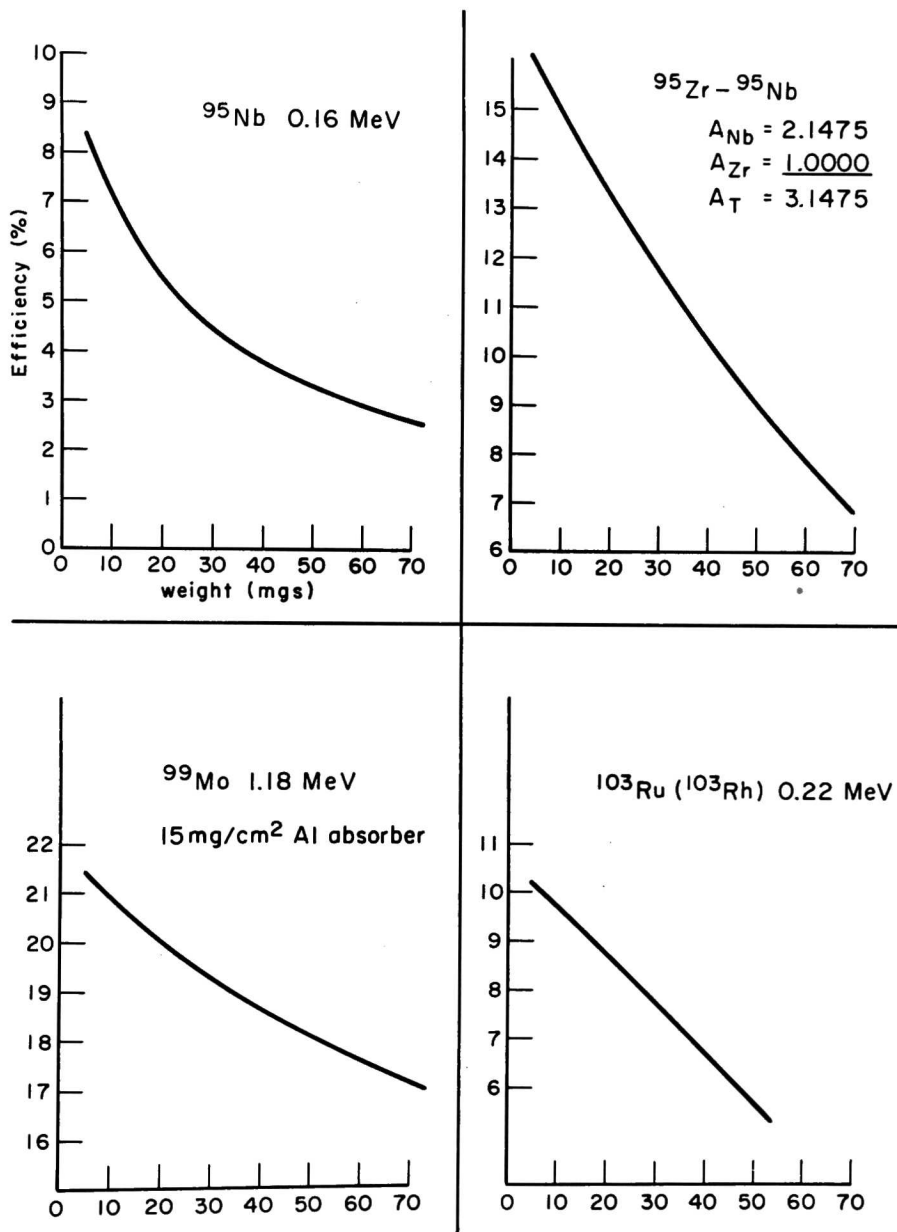


Fig. 6. β counting efficiency vs weight of precipitate. ANL Neg. No. 122-2272.

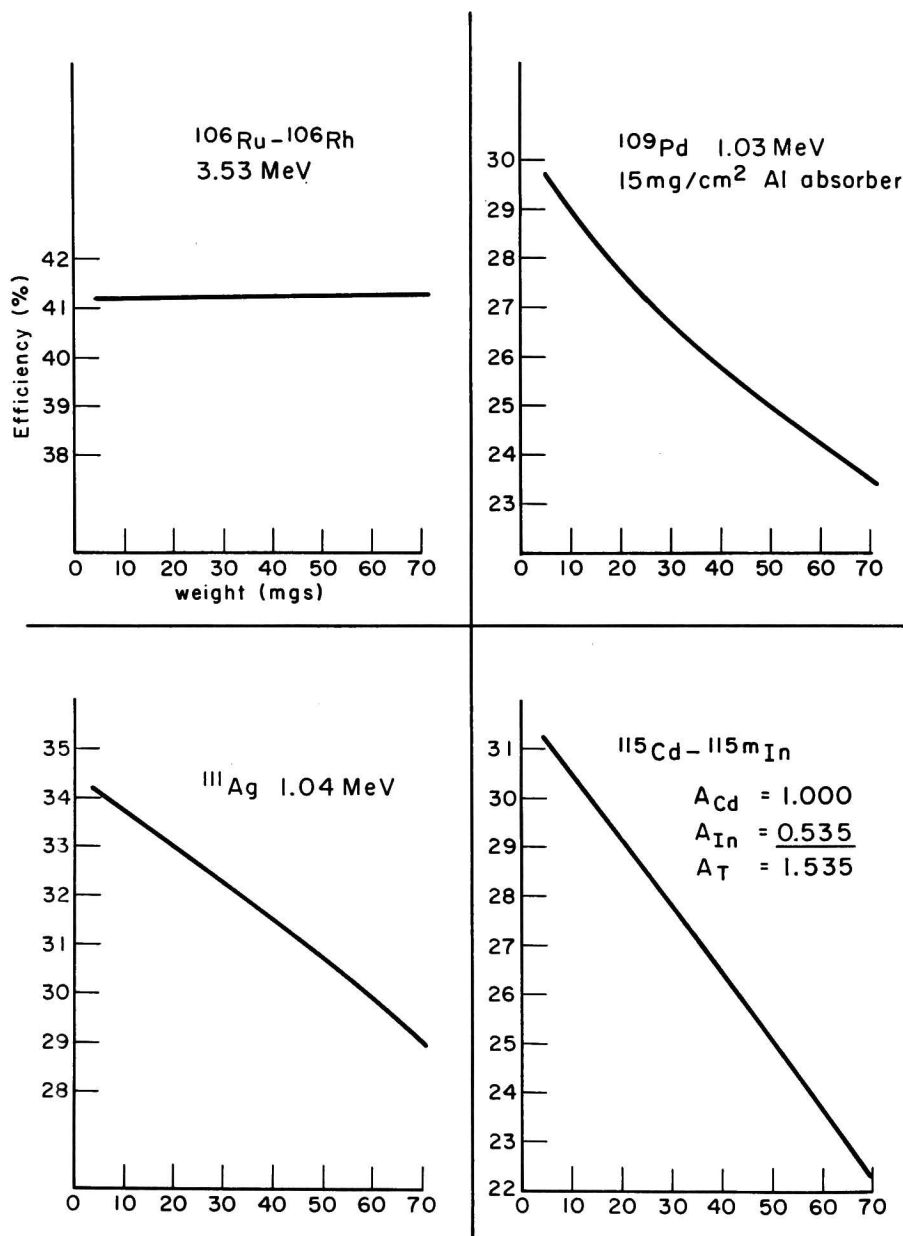


Fig. 7. β counting efficiency vs weight of precipitate. ANL Neg. No. 122-2270.

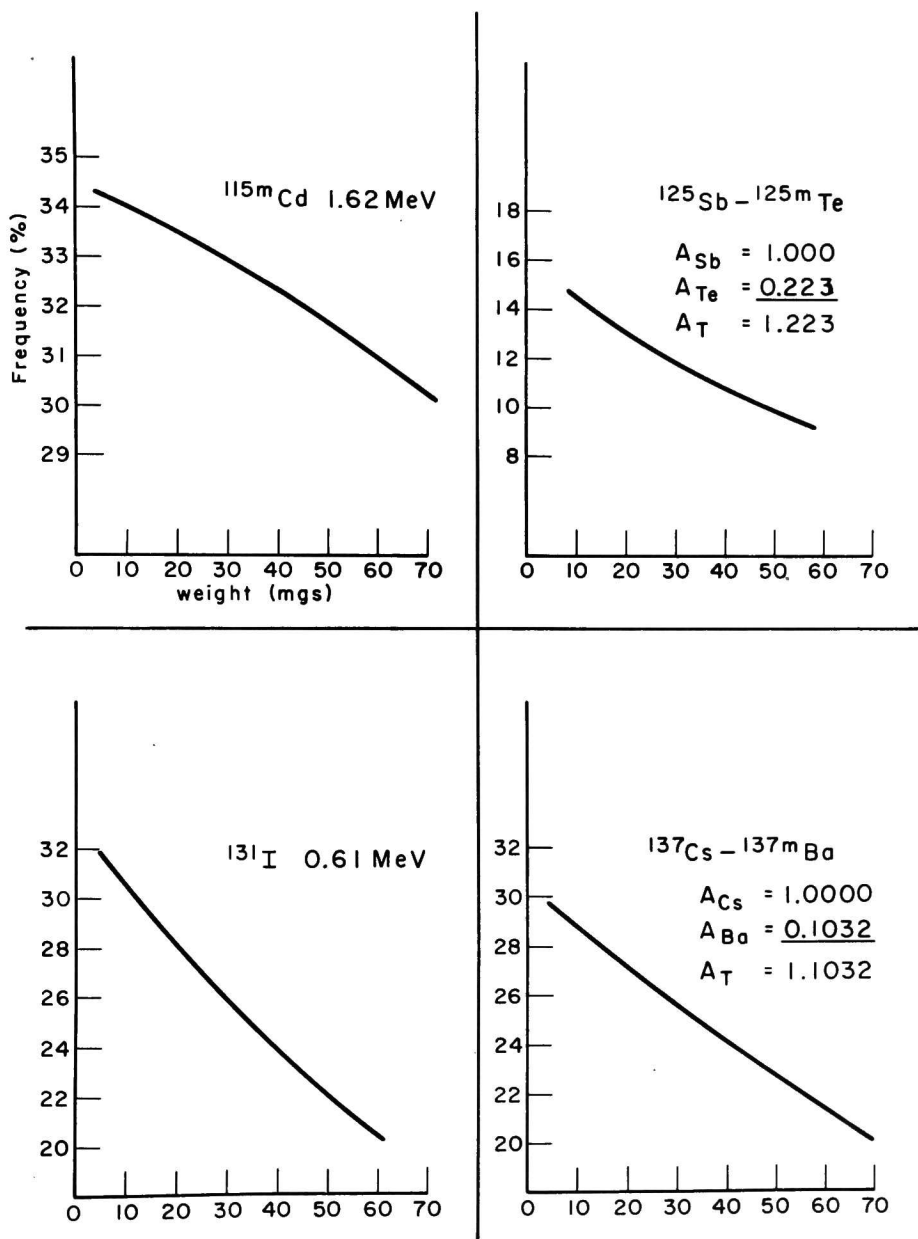


Fig. 8. β counting efficiency vs weight of precipitate. ANL Neg. No. 122-2268.

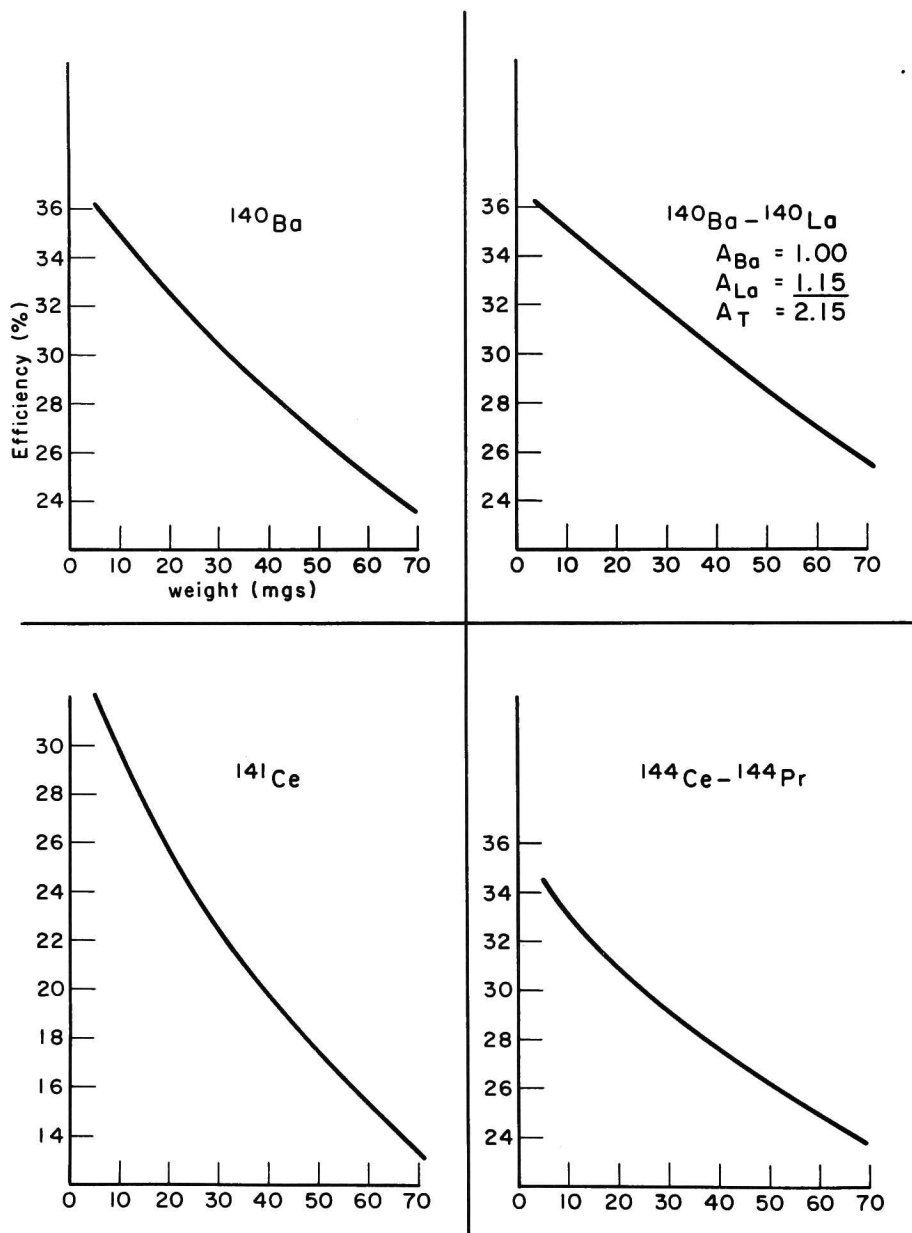


Fig. 9. β counting efficiency vs weight of precipitate. ANL Neg. No. 122-2264.

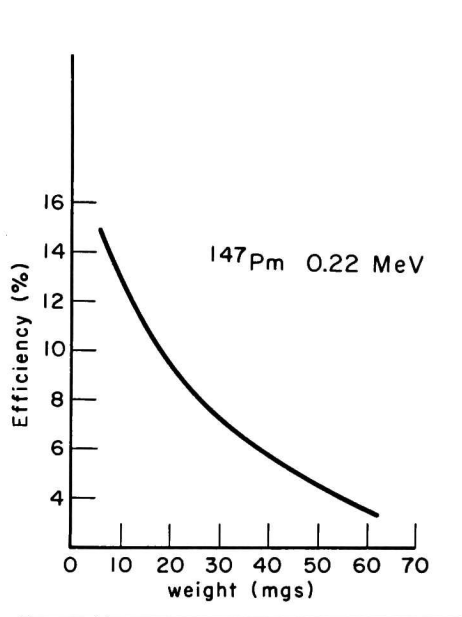


Fig. 10. β counting efficiency vs weight of precipitate.
ANL Neg. No. 122-2261.

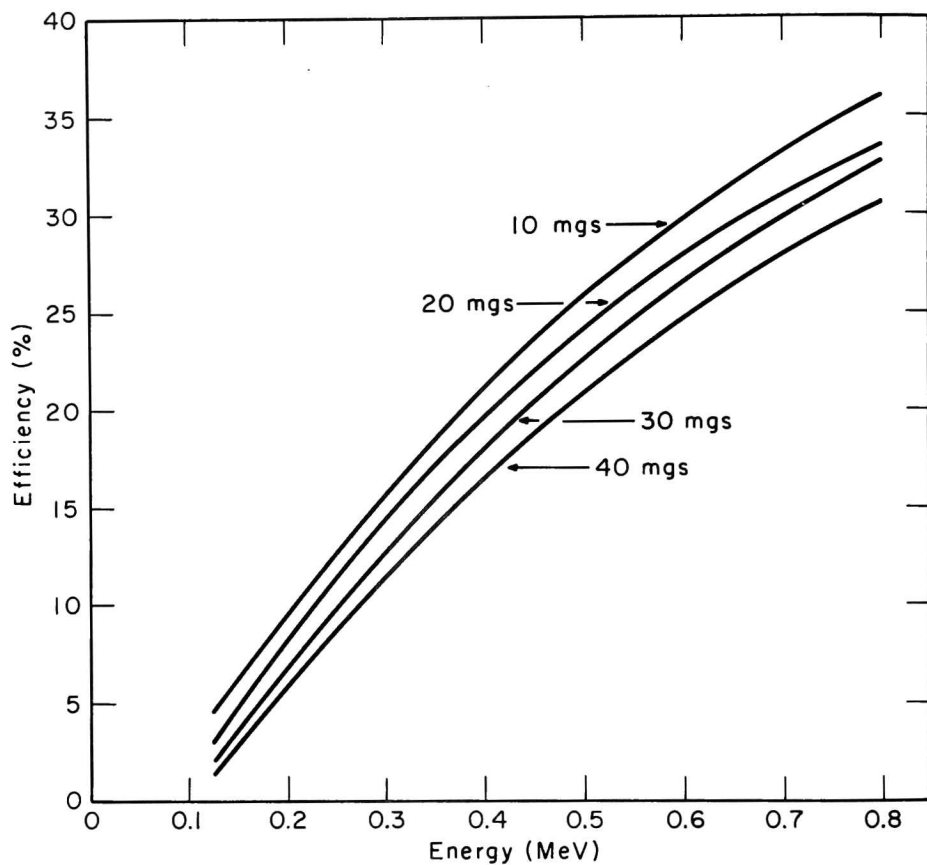


Fig. 11. β counting efficiency vs energy (.1 to .8 MeV) for 10, 20, 30, and 40 mg precipitates. (Samples on stainless steel discs covered with 1 mg/cm^2 mylar foil.) ANL Neg. No. 122-2265.

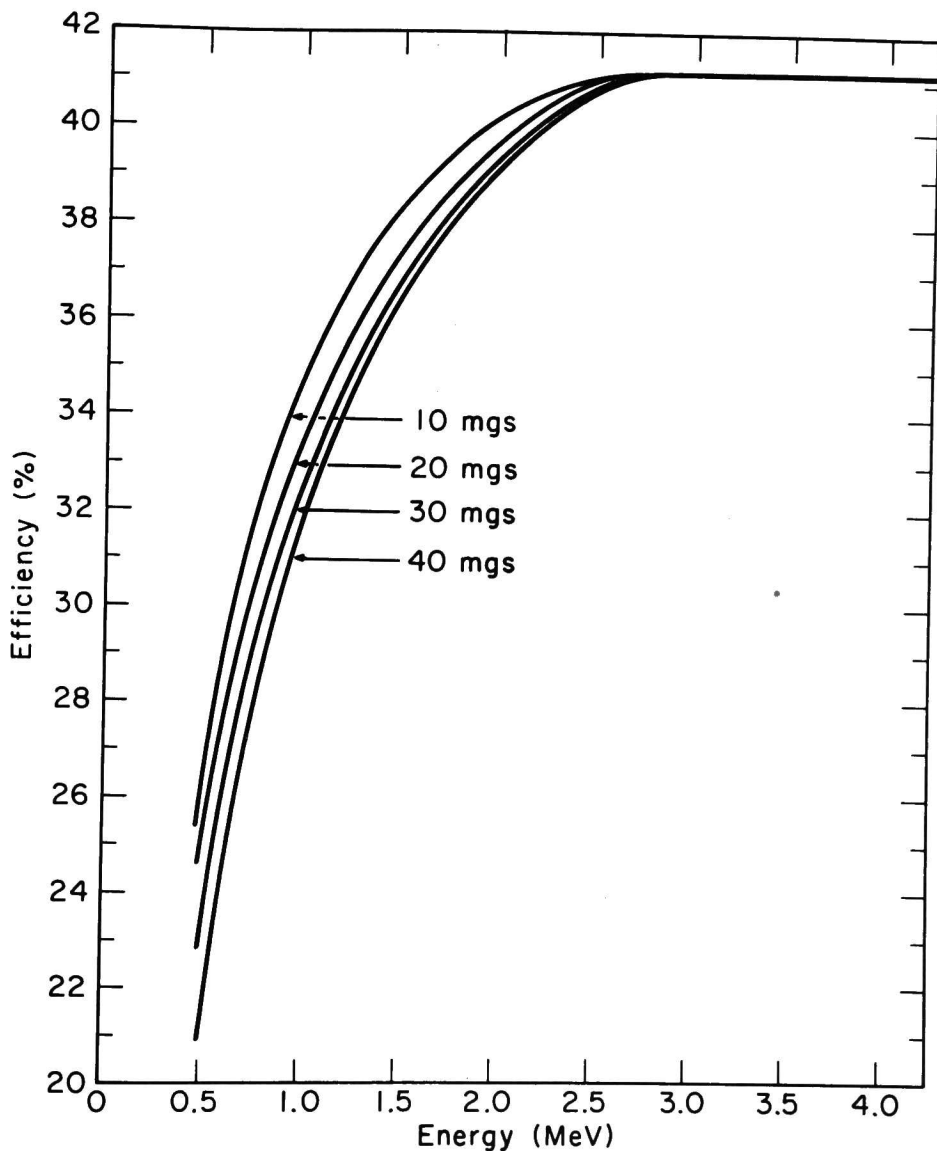


Fig. 12. β counting efficiency vs energy (0.5 to 4.0 MeV) for 10, 20, 30, and 40 mg precipitates. (Samples on stainless steel discs covered with 1 mg/cm² mylar foil.) ANL Neg. No. 122-2269.

of the 2π counter for that particular radionuclide. A series of 14 radionuclides ranging in energy from 40 keV (^{106}Ru) to 3.5 MeV (^{106}Rh) indicated a constant counting efficiency of $(78 \pm 1)\%$ for weightless samples on platinum backing.

The radioactive purity of radiochemically separated radionuclides is usually established by following its decay over several half lives. When mixtures of radionuclides are encountered in the same sample the decay curve can be analyzed by subtracting out the individual activities, one at a time. For those cases in which the half lives of the involved activities are not sufficiently different from each other to permit an accurate subtraction, a technique suggested by W. F. Biller (Ref. 15) can be used.

The total activity (A) of any sample at any time (t) can be expressed as a sum of exponential terms;

$$A = c_1 A_1^0 e^{-\lambda_1 t} + c_2 A_2^0 e^{-\lambda_2 t} \quad (1)$$

where A_1^0 and A_2^0 are the initial disintegration rates, λ_1 and λ_2 are the decay constants and c_1 and c_2 are the counting efficiencies. Equation (1) can be rearranged into the following form;

$$Ae^{\lambda_2 t} = c_1 A_1^0 e^{(\lambda_2 - \lambda_1)t} + c_2 A_2^0 \quad (2)$$

A plot of $Ae^{\lambda_2 t}$ against $e^{(\lambda_2 - \lambda_1)t}$ will give a straight line whose slope is $c_1 A_1^0$ and whose intercept on the $Ae^{\lambda_2 t}$ axis is $c_2 A_2^0$.

APPENDIX C. Gamma Counting Procedures

Lithium drifted germanium (Ge-Li) detectors have added a new dimension to the use of γ ray analysis as a method of quantitatively establishing the amount of a specific radionuclide present in a sample.

The Ge-Li detectors used in our work have been calibrated for overall efficiency using International Atomic Energy Agency (IAEA) standards, National Bureau of Standards (NBS) standards and some standards prepared by ourselves using beta-gamma coincidence counting techniques. All three calibrations checked each other within experimental error. A typical efficiency curve for point sources is shown in Figure 13. The gamma ray spectra obtained from the Ge-Li detectors are analyzed with an IBM-360/75 computer using the GAMMANAL program developed by R. Gunnick and J. B. Niday (Ref. 17) and modified by S. Kaufman of this Laboratory.

The excellent resolution (~ 2 keV full-width at half-maximum for the 1024 keV ^{60}Co line) of the Ge-Li detectors makes possible the determination of a large number of photopeaks in a single sample. Unfortunately the photopeaks analyzed in this manner must be of comparable abundance (within a factor of about 10) or they become lost in the Compton continuum. Hence it is necessary to do some chemical separations in order to obtain complete analysis of mixed fission product samples.

In Table 2 are listed the properties of the various fission product species which have been found suitable for determination by

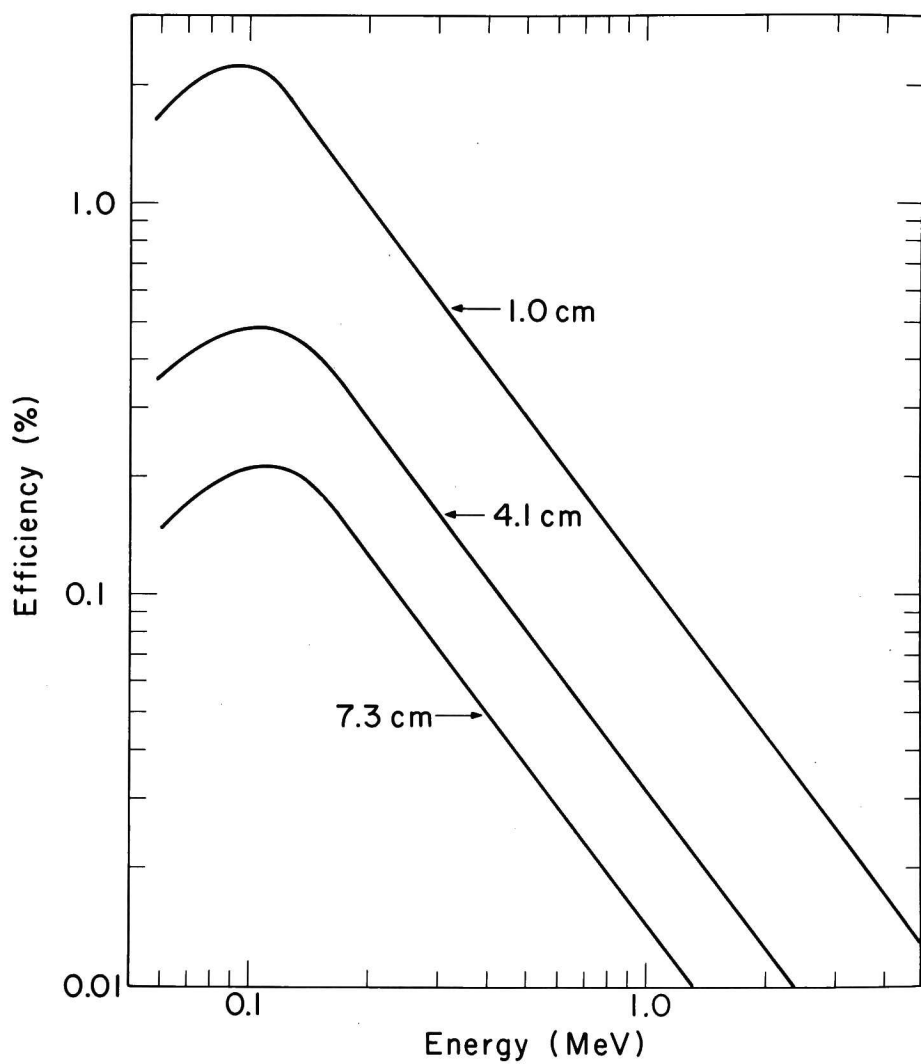


Fig. 13. Full energy peak efficiencies for a 17 cm³ coaxial GeLi detector with a 1.0 gm/cm² aluminum window. (Source to detector distances indicated.) ANL Neg. No. 122-2260.

Table 2. Decay Properties of Fission Product Nuclides

Isotope	Half-Life	Principal γ		Secondary γ	
		Energy (keV)	Abundance %	Energy (keV)	Abundance %
^{75}Ge	82.8 m	265	10		
^{77}Ge	11.3 hr	264	100	416	49
^{77}As	38.7 hr	239	1.4		
^{78}Ge	1.47 hr	277	94	294	6
^{78}As	1.52 hr	614	31	695	10
^{83}Br	2.4 hr	521	1.4		
^{85}Kr	4.48 hr	151	76	305	13.5
^{87}Kr	76.3 m	403	48	846	7.3
^{88}Kr	171.6 m	196	35	898	18
^{91}Sr	9.75 hr	750	23	1024	33
^{92}Sr	2.71 hr	1384	90	431	3.3
^{92}Y	3.53 hr	934	13.7	1405	4.7
^{93}Y	9.56 hr	267	7.2	947	2.0
^{95}Zr	65.5 d	724	43	757	54.6
^{95}Nb	35.1 d	766	99		
^{97}Zr	17.0 hr	743	93.3		
^{97}Nb	73.6 m	658	99		

Table 2 (Contd.)

⁹⁸ Nb	51 m	787	95	722	77	¹²⁵ Sn
⁹⁹ Mo	66.7 hr	740	14	181	97.6	¹²⁵ Se
⁹⁹ Tc	6.03 hr	140.3	90	142.3	9	¹²⁶ Se
¹⁰³ Ru	39.6 d	497	90	610	5.4	¹²⁶ mS
¹⁰⁵ Ru	4.44 hr	724	44.5	469	17.5	¹²⁷ Sn
¹⁰⁵ Rh	35.9 hr	319	19.6	306	5.4	¹²⁷ mS
¹⁰⁶ Ru	367 d	512	20.5	622	9.8	¹²⁷ Se
¹⁰⁹ Pd	13.47 hr	88	3.8			¹²⁸ Sn
¹¹¹ Ag	7.47 d	342	4.6			¹²⁸ Se
¹¹² Pd	20.12 hr	617	43	1388	5.3	¹²⁸ mS
¹¹² Ag	3.13 hr					¹²⁹ Se
¹¹³ Ag	5.3 hr	298	8.2	316	1.12	¹²⁹ Te
¹¹⁵ Cd	53.4 hr	528	27.5	492	8.1	¹²⁹ mS
¹¹⁵ mCd	44.6 d	934	2			¹³¹ I
¹¹⁷ Cd	3.4 hr	273	19.9	345	12.5	¹³² T
¹¹⁷ mCd	2.5 hr	1066	10.6	1235	25.2	¹³² I
¹¹⁷ In	44 m	159	86.7	553	99.7	¹³³ I
¹¹⁷ mIn	1.94 hr	315	21	159	17	¹³³ X
¹¹⁸ Cd	50.3 m	1220	100	525	14	¹³³ mS
¹²³ mSn	40 m	160	100			¹³⁴ P

Table 2 (Contd.)

^{125}Sn	9.65 d	1089	4.3	1066	8.9
^{125}Sb	2.73 yr	428	30.4	601	18.1
^{126}Sb	12.4 d	415	88	721	58
$^{126\text{m}}\text{Sb}$	19 m	666	100	695	100
^{127}Sn	2.12 hr	1114	29.2	1096	14.9
$^{127\text{m}}\text{Sn}$	4.4 m	491	100		
^{127}Sb	93 hr	685	36.8	473	24.8
^{128}Sn	59 m	482	66	557	18
^{128}Sb	8.9 hr	743	100	754	100
$^{128\text{m}}\text{Sb}$	11 m	314	95	743 [*]	100
^{129}Sb	4.35 hr	813	42.6	915	18.5
^{129}Te	69 m	460	100	487	19
$^{129\text{m}}\text{Te}$	33.5 d	460	100	487	19
^{131}I	8.04 d	365	82	637	6.8
^{132}Te	78 hr	228	88	668	98
^{132}I	2.28 hr	668	98	773	75
^{133}I	21 hr	530	87	875	4.5
^{133}Xe	5.29 d	81	35	79.6	1.6
$^{133\text{m}}\text{Xe}$	2.26 d	233	14		
^{134}Te	41.8 m	211	25	767	27

Table 2 (Contd.)

^{134}I	53.2 m	847	95.7	884	64.
$^{134\text{m}}\text{I}$	3.56 m	272	79		
^{135}I	6.7 hr	1261	29.1	527	11.
^{135}Xe	9.17 hr	250	92	609	2.
$^{135\text{m}}\text{Xe}$	15.6 m	527	80		
^{137}Cs	30.2 yr				
$^{137\text{m}}\text{Ba}$	2.55 m	662	85.1		
^{138}Cs	33.4 m	1436	75	1010	28.
^{139}Ba	82.7 m	166	73		
^{140}Ba	12.8 d	537	34	163	8.
^{140}La	40.2 hr	1596	96	487	46.
^{141}Ce	32.5 d	145	48		
^{142}La	95.4 m	641	47.5	2398	14.
^{143}Ce	33 hr	293	41.3	665	5.
^{144}Ce	284 d	134	10.8		
^{145}Pr	5.98 hr	675	45	747	47
^{147}Nd	11 d	91	14.9	531	9
^{149}Nd	1.8 hr	211	23	114	16
^{149}Pm	53.1 hr	286	2		
^{151}Pm	28 hr	340	21	168	7.
^{153}Sm	46.4 hr	103	28		
^{155}Eu	5 yr	86.5	32	125	20

Table 2 (Contd.)

^{156}Sm	9.4 hr	87.6	30	204	20
^{156}Eu	15.2 d	1230	8.7	812	9
^{157}Eu	15.2 hr	413	27	623	6
^{159}Gd	18 hr	364	10		
^{161}Tb	6.9 d	75	10		
^{166}Dy	81.5 hr	82	12		
^{167}Ho	3.1 hr	321	27		

gamma ray analysis using Ge-Li detectors. The data in this table have been taken from Ref. 18. A better knowledge of the abundance figures for many of the nuclides listed in Table 2 would greatly improve the reliability of the determinations of these nuclides by this technique. This table includes only the primary and secondary gamma rays for each isotope. For a complete listing of the gamma rays refer to Ref. 18.

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